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OXIDATION OF THIOSALT-CONTAINING EFFLUENTS

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by

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ABSTRACT

Room temperature ozonation of thiosalt-containing effluents was explored on a small laboratory scale to generate data for comparison with other potential treatment processes. The high oxidation potential of ozone (E° = 2.07 V) resulted in very fast and non-selective kinetics. The overall reaction mechanism is influenced by many parameters such as pH, solution composition, O_3 flow rate and oxygen concentration that result in observed O_3/S_2O_3 mol ratios of 1.6/1 to >5/1.

Implications for practical applications and for future fundamental experiments are discussed, and capital and operating cost estimates are derived using published literature data. For treatment of typical BMS milling effluents, the operating and maintenance costs of $4.1 M/a for ozonation compare favourably with similar cost estimates for oxidation with hydrogen peroxide or chlorine. The estimated capital cost of $22.5 M and ozone output requirements of up to 6000 t/a, however, are not considered to be practical.

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OZONISATION DES EFFLUENTS CONTENANT DES THIOSELS

par

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RÉSUMÉ

Une étude à l'échelle expérimentale portant sur l'ozonisation à température ambiante des effluents contenant des thiosels a été faite en laboratoire afin d'obtenir des données pour fins de comparaison à d'autres données compilées à partir des différentes méthodes de traitement envisagées. Le potentiel élevé d'oxydation de l'oxone (E° = 2,07 V) a donné lieu à une cinétique très rapide et non sélective. De nombreux paramètres, tels le pH, la composition de la solution, le débit de O_3 et la concentration d'oxygène influent sur le mécanisme de réaction global, ce qui produit des rapports moléculaires observables du O_3/S_2O_3 de 1,6/1 à > 5/1.

Ce rapport fait état des difficultés qui se présentent au niveau des applications pratiques et des expériences fondamentales futures et un devis estimatif des coûts des immobilisations et de fonctionnement tiré de données publiées antérieurement. Des coûts de fonctionnement et de maintenance au montant de 4,1 millions de dollars ayant trait au procédé d'ozonisation dans le traitement des effluents caractéristiques d'une usine de broyage de la BMS se comparent favorablement à un devis estimatif semblable des coûts de traitement par oxydation avec du peroxyde d'hydrogène ou du chlore. Le coût des immobilisations prévu de 22,5 millions de dollars et les exigences relatives à une production d'ozone pouvant atteindre 6000 t/a ne semblent toutefois pas favoriser la réalisation de ce traitement.

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INTRODUCTION

Ozone is one of the most powerful and non-polluting chemical oxidants (E^o = 2.07 V; products oxygen and water), but is also one of the most expensive to generate in large quantities. Ozone generally is produced by high voltage electrical discharge (15,000-20,000 V) in a dry oxygen-containing carrier gas stream. The ozone yield is a sensitive function of many parameters, such as the gas flow, pressure and temperature, oxygen concentration, moisture content (dew point), electrical power setting, condition of the discharge tubes, etc. (1). The resulting O_3 content of the gas never exceeds a few percent.

Basic reduction-oxidation considerations predict an overall reaction of ozone with thiosulphate involving the transfer of eight electrons, according to:

$$S_2O_3^{2-} + 4O_3 + H_2O = 2SO_4^{2-} + 2H^+ + 4O_2 \qquad (1)$$

Previous workers (2-4) have suggested that free oxygen in the carrier gas can also be involved in the oxidation step, to yield overall equations of the following form:

$$2S_2O_3^{2-} + 5O_3 + 2H_2O = 4SO_4^{2-} + 7/2\ O_2 + 4H^+ \qquad (2)$$

$$5S_2O_3^{2-} + 2O_3 + 7O_2 + 5H_2O = 10SO_4^{2-} + 10H^+ \qquad (3)$$

In view of the complexity of the chemistry of even the individual thiosalt and ozone systems, the contradictions among literature citations are not surprising, especially considering the simplicity of the ozone generating and metering systems generally employed (1,4,5). Fundamental chemical considerations predict the standard ozone half reaction to be:

$$O_3 + 2H^+ + 2e^- = O_2 + H_2O \qquad E^o = 2.07\ V \qquad (4)$$

Any deviation of the stoichiometry expected from equation (1) would be due to the participation of molecular oxygen in oxidizing a reaction intermediate such as SO_3^{2-}.

To complete the comparison of several potential methods for the destruction of thiosalts in milling effluents (6-8), the present study was undertaken to define some realistic ozone requirements for thiosalt oxidation as a basis for a proposed cost comparison study (7). Objectives were to examine the process parameters on a laboratory scale to generate data for comparative evaluation purposes, and to expand our knowledge of the chemistry of thiosalt oxidation (9,10). The results of the tests and insights gained during the experiments, however, also proved to be interesting from a fundamental chemical point of view, and may be worth pursuing in a more academic type study.

EXPERIMENTAL

The basic experimental set-up is shown schematically in Figure 1. High purity air or oxygen at 180 to 225 kPa was dried and metered into a Welsbach model T-816 Ozone generator (1). The output gas stream was diverted to one of two trains of absorption flasks by means of a Teflon/glass three-way valve. This allowed multiple tests with thiosalt oxidation and/or O_3 output calibration measurements without changing solutions. Preliminary tests showed that output gas flow rates as measured by the rotameters built in the generator would fluctuate significantly as a function of the absorption train resistance. Since the ozone output was measured indirectly (see below), the maintenance of more constant operating conditions was deemed important. Therefore, the feed gas flow was automatically controlled below 1 L (STP)/min by means of a Mattheson Model 8250 Electronic mass flow controller. This

stabilized the ozone gas output to the extent that its flow rate and delivery pressure were more easily controlled and maintained.

Each absorption train consisted of a primary absorber containing 400 to 500 mL of solution and a small exit gas scrubber containing 100 mL of 2% KI solution. The primary absorber contained 2% KI solution for O_3 output calibration, or test solutions for determining redox stoichiometries. To facilitate analytical procedures the exact starting volumes must be known, and were determined by careful additions from calibrated burettes. Faster and more accurate determinations would require the use of a dual range electronic balance.

The ozone output was determined by the amount of I_2 produced upon acidification of the absorbing KI solutions. The chemistry of the reactions involved is by no means simple (2) although an overall reaction of the following type is assumed:

$$O_3 + 2I^- + 2H^+ = O_2 + I_2 + H_2O \qquad (5)$$

A white mist (2) generally formed over the KI solutions, and after extended periods of time solid I_2 formed within the fritted glass spargers used. In typical plant solutions, oxidation of iron(II) to iron(III) resulted in the formation of a crystalline precipitate, and test solutions generally had a distinct ozone smell.

Thiosalt determinations were conducted by means of standard iodometric and $HgCl_2$ acidimetric methods (6,8-10). For synthetic thiosulphate solutions the results of both methods were identical if care was taken in adjusting the pH of the $HgCl_2$ acidimetric reagents to the pH 4.3 value used in the titrations. In actual plant solutions, interferences by iron and other matrix components might have been present, and were not corrected for. Although speciation between the thiosalts and other reduced sulphur species is not

regarded to be critical in these exploratory studies, the comparison of iodometric (thiosulphate and sulphite) and the $HgCl_2$ acidimetric values (total thiosalts) yield useful information, as would the accurate determination of sulphate.

The oxidation results were still subject to large experimental errors, and few possible improvements are recommended for any future fundamental ozonation studies. In combination with the mass flow controller, direct determination of the O_3 concentration in the gas streams by spectrophotometric means would be preferred over the present iodometric method. Errors due to residual ozone in the feed lines could be eliminated by using a four-way valve with inert gas or air purge connections. The lower gas flow rates made possible by the use of the mass flow controller were compatible with the size of the absorbtion flasks as evidenced by the high O_3 utilization values corresponding to low O_3 levels in the exit gas from the primary absorbtion flask. Monitoring of pH or e.m.f., automatic pH control and thermostatting would be essential. The O_3 output is best controlled by variation of either the power input or the flow rate and feed gas composition to the generator.

RESULTS AND DISCUSSION

PRELIMINARY TESTS

Initial tests to assess the experimental reproducibility and parametric dependances were conducted with synthetic solutions of thiosulphate in deionized water. The results are summarized in Table 1 according to the increasing degree of S_2O_3 conversion. The ozonation output is difficult to predict from equipment control settings, so that a great deal of uncertainty is introduced into the end results even though the analytical titrations were very reproducible.

The low conversion test with air was most unreliable, but was of interest because H_2S formation was unexpected in the presence of a strong oxidizing agent such as O_3. Four intermediate conversions were achieved with a wide variation in experimental conditions, but the end results were surprisingly similar and no persistent trends emerged. In the last test, unexpectedly high ozone output under normal conditions resulted in nearly complete $S_2O_3^{2-}$ conversion to sulphate (nearly identical results for iodometry and $HgCl_2$ acidimetry were obtained through these preliminary tests).

It was concluded that the oxidation of thiosulphate with ozone is almost instananeous and is, therefore, not very selective. Excluding the first test, the average ozone requirements were 3.3 mol per mol of thiosulphate. The higher O_3:S_2O_3 mole ratio of 5:1 in the last test might indicate less participation of molecular oxygen at higher ozone output rates.

MULTIPLE OXIDATION TESTS

It was argued that much of the experimental uncertainty could be removed by repeating alternate O_3 oxidation and output calibration measurements in rapid succession without changing the basic instrument settings. After initial warm-up and stabilization of the ozone generator, the gas output stream was alternatively switched at timed intervals between two absorption trains by means of a three way valve. This allowed sampling of the oxidized solutions before continuation with the next exposure to ozone, as well as monitoring of the instrument settings, provided the ozone output was not too high and the exposure time intervals were >2 min.

Table 2 shows that the ozone output with air as the ozonation feed gas is quite unstable, and that high overall O_3:S_2O_3 mol ratios of about 5.5:1 are indicated. The O_3 output with oxygen as the feed gas appeared to

be more stable and reduced the overall O_3:S_2O_3 requirements to about 2.5:1, but the retention times were too short to allow precise measurements. This was remedied by selecting a lower power setting of 90 V, combined with a lower oxygen flow rate of 0.5 L/min, as shown for the test in Table 3. The ozone output was fairly stable except during the initial and final absorption periods. It was concluded that the O_3 production was essentially constant, but that some experimental shortcomings remained that might be resolved by following the recommendations outlined in the experimental section. Therefore, the differential O_3/S_2O_3 mol ratios during the stable portions of the test are to be used rather than the overall results, and it appears that the ozone requirements are between 3 and 4 mol per mol of thiosulphate.

The above test results with air and oxygen are also shown in Figure 2. The rates of ozone generation and thiosulphate oxidation seem directly related, indicative of a very fast reaction. In the test with oxygen, the linear decrease of the thiosulphate concentration reflects the relatively constant ozone production rate of about 0.7 mmol/min. The overall O_3/S_2O_3 mol ratio with air appears to be about double the requirement with oxygen.

TESTS WITH REAL MILLING EFFLUENTS

Preliminary tests were also conducted with samples of thickener overflow solution received from Brunswick Mining and Smelting (BMS) that was spiked with additional thiosulphate. The background matrix analysis of this effluent was given previously (10), and Table 4 summarizes the relevant details of the ozonation tests. When a typical sample of BMS solution, spiked with 10 mmol/L thiosulphate, was ozonated for 3 min, the product solution (pH 2.8) contained mostly thionates, some thiosulphate or sulphite,

and traces of elemental sulphur. The O_3/S_2O_3 mol ratio was only 1.62/1, indicating that acid decomposition might have contributed significantly to the thiosalt elimination.

Another BMS solution sample was again spiked with 10 mmol/L thiosulphate, made alkaline (pH 11.5) with 1 M NaOH, and preoxidized by oxygen sparging to precipitate ferric hydroxide. The decanted solution was ozonated for 6 min, resulting in more complete thiosalt removal, again with a low O_3/S_2O_3 mol ratio of 1.67/1. The final pH was about 4 and traces of colloidal S^o were again observed. The solution was again decanted from additional amounts of crystalline ferric material, spiked with more thiosulphate and a larger amount of NaOH (pH 12.5), and subjected to a multiple oxidation test, summarized in Table 4 and shown in Figure 3. The O_3 output rate was fairly constant throughout the test and the average value of 0.6 mmol/min was used for all the intervals, including the initial 3 min which characteristically yielded a lower O_3 output value of about 0.45 mmol/min. During this initial period, the thiosalt concentration actually increased. This probably indicates oxidation of colloidal elemental sulphur producing thiosalt intermediates at the high intial pH, either directly or via reaction with other thiosalts or sulphite; e.g.:

$$1/8\ S_8^o + SO_3^{2-} = S_2O_3^{2-} \qquad (6)$$

Further oxidation proceded very much as for the synthetic solutions (compare Figures 2 and 3), and the overall O_3/S_2O_3 mol ratio returned to "normal" values between 3/1 and 4/1.

It is therefore postulated that the ozonation mechanism in real effluents is very much pH dependent. Under "controlled" conditions; e.g., in buffered or pH controlled solutions with low to moderate O_3 in oxygen flows, the anticipated O_3/S_2O_3 mol ratio will be close to 3/1. In more acidic solutions

or with higher O_3 flow rates resulting in low local pH values, the O_3/S_2O_3 mol ratio may drop to about 1.6/1 due to acid decomposition of thiosulphate. Byproduct S^o and H_2S are likely to be formed under these conditions. Since O_3 requirements are expected to double when O_3/air mixtures are used, the most economic industrial application would provide pure O_2 feed gas for ozonation, with recycle of residual O_2 streams via O_3 decomposition reactors, dryers and compressors to the ozone generator (7). Table 5 summarizes some literature data on the capital and operating costs of large industrial ozonation plants. Treatment of typical BMS effluent containing 1000 mg/L S_2O_3 at a flow rate of 10,000 L/min (3,5) would require 5630 t/a ozone and 3470 t/a lime if equation (2) is used as a basis for estimation. Using a unit cost of $50/t of lime and the BMS cost estimate of Table 5, total capital and operating costs would be $22.5 M and $4.1 M/a, respectively.

CONCLUSIONS

The ozonation of thiosalt-containing effluents appears to be very fast and non-selective compared to oxidation with hydrogen peroxide or sodium hypochlorite. The ozonation mechanism was very dependent on the pH, solution composition, ozone flow rate and oxygen concentration even though the reactions occurred almost instantaneously. Thus, H_2S and S^o byproducts were formed under low pH conditions resulting in decreased molar O_3/S_2O_3 ratios. At intermediate pH values, thiosalts were detected in partially ozonated BMS plant solutions, whereas synthetic thiosulphate solutions were always oxidized directly to sulphate. At high initial pH values, elemental sulphur contained in aged or preoxidized BMS solutions was oxidized to thiosalts, but sulphur is a stable byproduct under low pH conditions as mentioned above.

It is therefore suggested that S^o oxidation could also have contributed to the higher ClO^- requirements for the alkaline chlorination of BMS solutions (10). whereas the S^o would have been stable in the acidic solutions used for the similar oxidation with hydrogen peroxide (8).

Test results indicated that the ozonation of thiosalt-containing effluents is technically simple, but quite expensive. Relatively low molar O_3/S_2O_3 requirements were observed (1.6/1 to 4/1) with oxygen as the feed gas. Uncertainties about the exact reaction stoichiometries were due mainly to experimental shortcomings such as indirect O_3 determination, fluctuations in O_3 output, lack of pH control, incomplete solution speciation, etc. These difficulties can be overcome, however, especially on a large industrial scale, and costing was based on the generally accepted equation (2) with requirements of 2.5 mol O_3 and 1 mol lime per thiosulphate equivalent.

Total cost estimates from published data yielded a $22.5 M capital and $4.1 M/a operating and maintenance costs. Although reagent costs are reasonable compared to $8.5 M/a for H_2O_2 (8) and $3.8 M/a for chlorine oxidation (10), the capital outlay is about one order to magnitude too large to be practical. The required ozonation plant would be about ten times larger than any existing facility.

In view of the fundamental insights gained by this series of thiosalt destruction studies (6-10 and references), it appears that chemical oxidation processes are viable and reliable, but very expensive. The best practical method might well consist of a combination of existing options. Future research should concentrate on developing such an integrated process, possibly incorporating catalysed acid decomposition and/or molecular oxygen reduction, as well as advanced flotation, liquid/solid separation and tailings management technologies.

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Table 1. Preliminary Ozonation Test Results

Standard Conditions: 400 mL synthetic $S_2O_3^{2-}$ test solutions, 0.9 L/min O_2 feed (mass flow meter), 100 to 110 V power setting, 5 to 10 min retention time, 140 to 155 kPa delivery pressure, initial pH $\geq$10, room temperature

% S_2O_3 conversion	O_3 output mmol/min	% O_3 utilized	S_2O_3 (mmol/L) start	end	O_3:S_2O_3 mol ratio
0.75*	0.15	83	10.26	10.18	(17:1)
30.6†	0.98	90	4.62	3.17	3.13:1
37.2†	1.13	74	4.77	2.99	2.34:1
47.8	0.97	99	9.5	4.95	3.14:1
50.0	1.09	99	9.0	4.50	2.81:1
>99	2.20	90	10.0	<0.1	4.97:1

* 1 L/min air, 90 V; initial pH 7.0; H_2S formation; 4 min retention time; minimal conversion and unreliable stoichiometry.

† no mass flow control, 1.35 and 2.1 L/min output gas flow, initial pH 5.2 and 11.0, respectively; 100 V power setting; 2 min retention time

Table 2. Multiple Oxidation Test with Air

Conditions: 400 mL synthetic thiosulphate solution, initial pH 11.5, room temperature, 0.9 L/min air feed, 150 kPa delivery pressure, 110 V power setting

Exposure time (min)	Differential O_3 output mmol/min	S_2O_3 concentration mmol/L	Calculated % S_2O_3 conversion	O_3:S_2O_3 mol ratio	
				diff.	total
0	-	10.735	0	-	-
2	0.4	10.108	6.3	3.45:1	3.45:1
4	0.79	(9.6)	9.2	8.49:1	5.71:1
6	0.39	9.424	13.0	12.2:1	6.58:1
10	0.192	(9.0)	19.0	5.05:1	5.62:1
19	0.19	8.132	25.5	5.59:1	5.01:1
21*	1.50	4.104	62.5	1.86:1	3.14:1
22.5*	1.38	1.824	83.4	0.88:1	2.17:1

* 0.9 L/min O_2 feed at 100 V, 150 kPa delivery pressure

Table 3. Multiple Oxidation Test with Oxygen

Conditions: 400 mL synthetic thiosulphate solution, initial pH 12.5, room temperature, 0.5 L/min O_2 feed, 146 kPa delivery pressure, 90 V power setting

Exposure time (min)	Differential O_3 output mmol/min	S_2O_3 concentration mmol/L	Calculated % S_2O_3 conversion	O_3:S_2O_3 mol ratio	
				diff.	total
0	-	9.88	0	-	-
2	0.40	8.21	17.5	1.2:1	1.2:1
4	0.60	7.15	28.5	3.78:1	2.19:1
6	0.66	6.13	38.6	3.92:1	2.65:1
12	0.68	2.38	76.5	2.95:1	2.79:1
16	0.68	0	100	2.61:1	2.83:1
20	0.66	0	100	n.a.	n.a.
22	0.74	0	100	n.a.	n.a.

Table 4. Tests with BMS Thickener Overflow

Time min	Analysis (mmol) S_2O_3	Analysis (mmol) S_2O_3*	pH	O_3 output mmol/min	% O_3 utilized	% S_2O_3 conversion	O_3:S_2O_3 mol ratio
0**	10.9	12.2	4	-	-	-	-
3	1.48	5.38	2.8	1.23	99	56.0	1.62:1
0**	8.9	9.1	11.5	-	-	-	-
6	0.15	0.99	4	1.04	76	89.1	1.67:1
0†	n.a.	8.8	12.5	-	-	-	-
3	n.a.	12.15	n.a.	∿0.6	99	0	(-1.2:1)
6	n.a.	10.82	n.a.	∿0.6	99	10.9	3.05:1
9	n.a.	9.24	n.a.	∿0.6	99	23.9	2.59:1
12	n.a.	7.73	n.a.	∿0.6	99	36.3	2.69:1
15	n.a.	6.71	∿4.5	∿0.6	99	44.7	3.93:1
18	n.a.	5.92	<4	∿0.6	99	51.2	4.46:1
21	n.a.	4.65	n.a.	∿0.6	89	61.7	3.37:1
24	n.a.	3.25	n.a.	∿0.6	n.a.	73.3	3.09:1

* Total thiosalts

** preliminary tests: ∿400 mL solution spiked with $Na_2S_2O_3$, 0.9 L/min, O_2 feed, 110 V, ∿147 kPa delivery pressure, room temperature

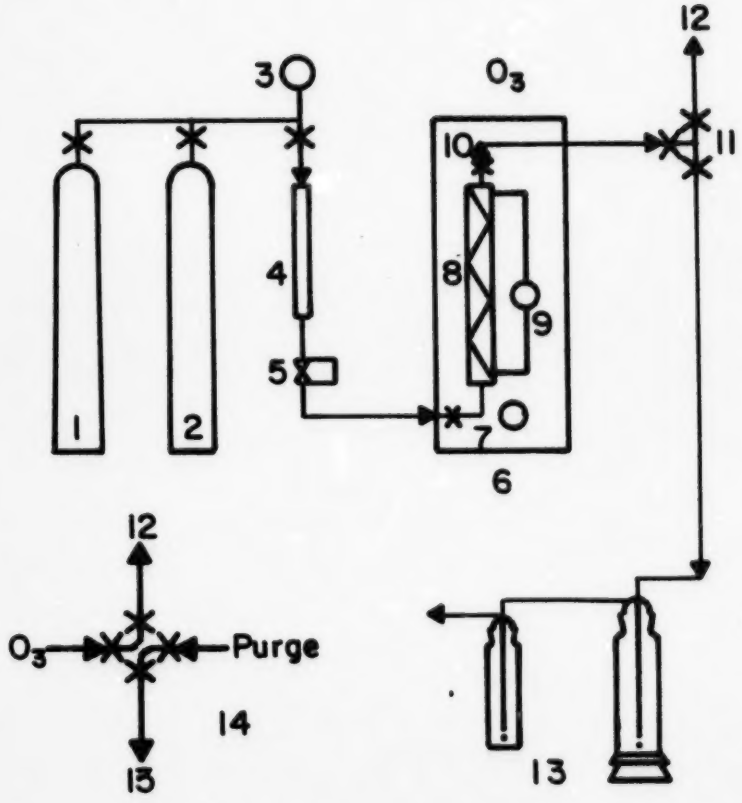
† multiple oxidation test: 400 mL BMS solution from preliminary test, spiked with 40 mL 0.1 M $S_2O_3^{2-}$ and 10 mL 1 M NaOH, 0.5 L/min, O_2 feed, 90 V, ∿148 kPa, room temperature

Table 5. Ozonation Cost Data

Source	Output capacity t/a	Installed capital cost $ per t/a	Operating & maintenance $/t*
Montreal Engineering Ltd., 1975 (11)	0.9 K	1.5 K	0.92 K
Tektronix Inc., 1978 (5)	0.03 K	5.1 K	0.74 K
French operators 1980 (12)	0.45 K	14 K†	0.875 K
Sherritt Gordon Mines Ltd., 1982 (13)	0.75 K	4.1 K	0.74 K
Degremont, 1984 (7)	0.83 K	6-9.6 K	0.39-0.77 K
BMS cost estimate, this work	5-10 K	4 K	0.7 K

* Assuming power costs of $0.035/kWh; costs can also be expressed in kWh/kgO_3.

† Estimated from total carrying charges, assuming a 10 year amortization period and a conversion rate of 7 francs per dollar



1 = O_2 cylinder
2 = Air cylinder
3 = Delivery pressure, P_1
4 = Drying tube
5 = Mass flow controller
6 = Ozone generator
7 = Generator pressure, P_2
8 = Discharge tube
9 = Voltage regulator
10 = Delivery flow regulator
11 = Three-way valve
12,13 = Absorption trains
14 = Proposed four-way valve arrangement

Figure 1. Apparatus

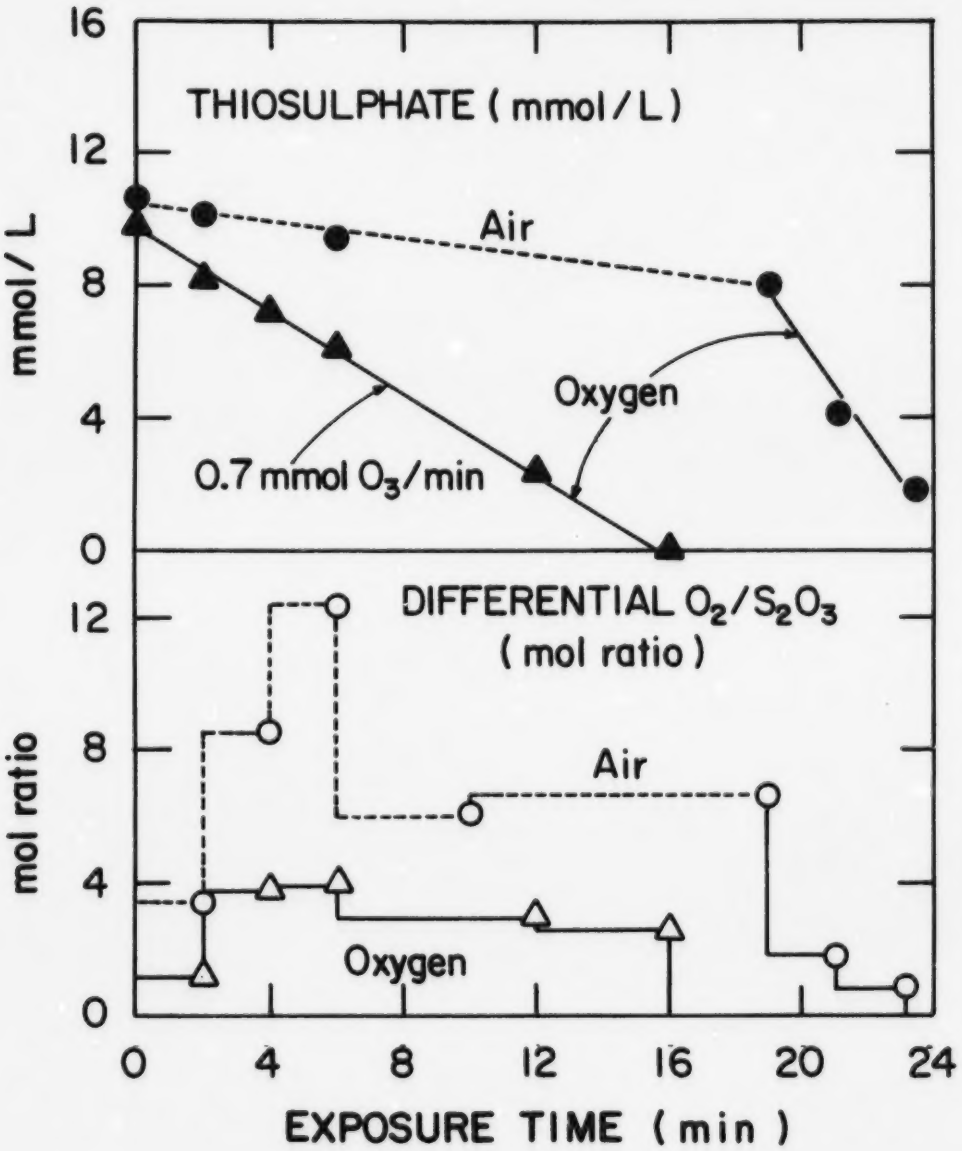


Figure 2. Multiple oxidation of synthetic solutions

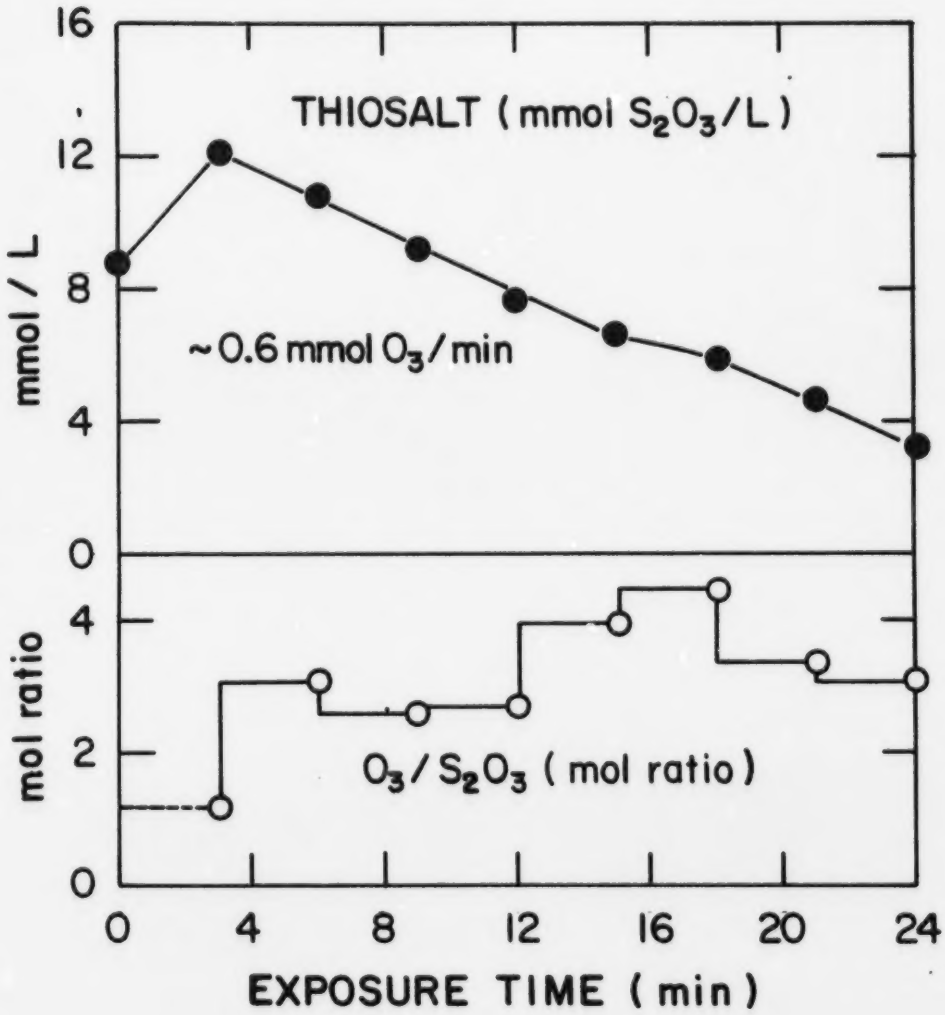


Figure 3. Multiple oxidation of BMS effluent